[0219] (9) Shut down

[0220] (a) Turn off all heaters and pumps, drain the knock out pots, switch feed ball valves to purge nitrogen through system being careful not to blow liquid out the top of the knock out pots to the vent line, drain knock out pots again when the lines appear to be mostly cleared of liquid and then leave nitrogen purge on during the rest of the cool down. Once cool (<60° C.), turn off nitrogen purge and the chiller.

EXAMPLE

[0221] A microchannel distillation unit comprises an assembly of two plates—a liquid plate and a vapor plate. The unit is schematically illustrated in **FIG. 43**. The plates are made out of Stainless Steel 316. The liquid plate has a channel for liquid flow while the vapor plate has a channel for vapor flow. The overall dimensions of liquid plate are 7.6 cm wide×1 cm thick×22.9 cm long. The overall dimensions of the liquid channel in the liquid plate are 2.9 cm width×1.0 mm height×14 cm long. The liquid removal structure is a 72×72 stainless steel mesh. The overall thickness of the mesh is 0.36 mm. The mesh is spot welded on the wall of the liquid channel. The liquid enters the device at one end of the liquid removal structure and exits the device at the other end of the liquid removal structure as shown in **FIG. 43**. At the liquid inlet, a manifold having the dimensions of 2.5 cm wide×2.5 mm high×2.5 mm deep is provided for uniform flow distribution over the entire width of the liquid removal structure. The distribution of liquid from the manifold to the liquid removal structure is through 6×1.5 mm diameter holes. At the outlet of the liquid, a fine mesh made from Pall Supramesh (stainless steel, 25 micron pore size) is placed. The fine mesh acts as a pore throat to prevent vapor breakthrough in to the liquid. The dimensions of the pore throat are 2.5 cm×2.5 cm×0.36 mm. The overall dimensions of vapor plate are 7.6 cm wide×0.7 cm thick×22.9 cm long. The vapor channel dimensions are 2.2 cm wide×0.5 mm height×11 cm long.

[0222] The plates are assembled together using bolts and nuts. A gasket is placed between the plates to prevent leakages to outside. The inlets and outlets for liquid and vapor are designed such that during the assembly of device, the liquid inlet and outlet are offset from vapor inlet and outlet by 1 cm.

[0223] The desired feed composition for liquid and vapor is achieved by mixing measured quantities of n-hexane and cyclohexane. Both n-hexane and cyclohexane are obtained from Sigma-Aldrich and have greater than 99% purity. Pressures are measured using a pressure tranducer (Manufacturer—NoShok Model No: 100-30-2-1-2-7) with accuracy of ±0.07 psi, range 0-30 psig. The temperatures are measured using RTD (Manufacturer—Omega, Model No: KMQSS-010U-18) with a temperature measurement range from −220° C. to 220° C. and accuracy of ±0.2° C. The flow composition is measured using Gas Chromatograph (Manufacturer—Agilent Technologies, Model No: 6890N) with temperature range from 4° C. to 450° C. The flow is supplied by syringe pumps (Manufacturer—Cole Parmer, Model No: 74900-00) with flow rate from 0.2 μL/hr to 500 mL/hr and accuracy of ±0.5%. The flow rate is measured with flow meters (Manufacturer—Cole Parmer, Model: EW-03268-09), full scale range of 1.75 ml/min with accuracy of ±2% of full scale.

[0224] The feed inlet composition, temperature, pressures and flow rate of the liquid stream are maintained at 83.9% n-hexane by mass, 67.6° C., 0.1 psig and 1.01 L ml/min respectively. The feed composition, temperature, pressures and flow rate of the vapor stream are maintained at 8.5% n-hexane by mass, 84.2° C., 0.1 psig and 1.00 L ml/min respectively. The heat loss from the system is reduced by placing the device in a furnace. The furnace space temperature is set at 55° C. The wall temperature of the device is controlled using strip heaters. The strip heaters are located 0.5 inch" above the vapor inlet location on both vapor and liquid plate. The voltage settings in the heaters are set such that the metal temperature near the vapor inlet is approximately 77° C. while the metal temperature near the liquid inlet is approximately 73° C. The steady state condition is maintained for 25 minutes before recording the outlet flow conditions and collecting outlet samples for composition analysis. The data is recorded using Data Acquisition System (Lab-view). The data is recorded every three seconds.

[0225] The outlet composition, temperature, pressures and flow rate of the liquid stream are maintained at 9% n-hexane by mass, 75.1° C., 0.1 psig and 1.01 L ml/min respectively. The outlet composition, temperature, pressures and flow rate of the vapor stream are maintained at 8.5% n-hexane by mass, 72.7° C., 0.1 psig and 1.00 L ml/min respectively. From the measured outlet composition and known inlet composition and flow rate, outlet flow rate is estimated. The experimental data is then used to develop a ChemCAD simulation to predict the number of equilibrium stages. The number of equilibrium stages for the device and operating conditions is 10 which gave an HETP of 0.5 inch.

[0226] Pore throat plays an important role in fluid separation. As a result of separation, heavier components are rich in liquid phase while lighter components are rich in vapor phase. The heavier components are removed from microchannels through dense structure called pore throat. The pore throat prevents vapor break-through into the liquid. The capacity of the microchannel and the phase separation is determined by the pore throat. Some examples of pore throat but not limited to, are—Pall Supramesh (25 microns pore size), 72×72 stainless steel mesh, liquid pool etc.

[0227] In PCT International Publication No. WO 03/049835 A1, which is incorporated herein by reference, it is disclosed that the Suratmann coefficient in the microchannel determines the efficiency of liquid and vapor mixture phase separation using liquid removal structures (wicking structures). It is disclosed that a good phase separation of liquid and vapor mixture is obtained for ratio of gas phase Reynolds number to liquid phase Reynolds number greater than (4500)×(Suratmann number)$^{-0.67}$. However, the pore throat also plays an important role in the efficiency of phase separation in microchannels using liquid removal structures. Experiments conducted with air-water mixture show the effect of pore throat in phase separation. A hardware as described in the example in WO 03/049835 A1, is fabricated and tested for air-water mixture separation. One modification is made in the arrangement of liquid removal structures (wicking structures). The arrangement of Pall Supramesh and 72×72 mesh are reversed as shown in **FIG. 44**. The orientation of the device is horizontal. The air-water mixture is made by mixture water flowing at 80 ml/min and air flowing at 0.35 SLPM. The suction heat for the liquid is 25.4 cm. The ratio of gas phase Reynolds number to liquid phase